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**YAG:Ce<sup>3+</sup>和 YAGG:Ce<sup>3+</sup>荧光粉及透明陶瓷的  
制备与性能研究**

**Synthesis and properties of YAG:Ce<sup>3+</sup> and YAGG:Ce<sup>3+</sup> phosphors  
and transparent ceramics**

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## 摘要

由于白光 LED 具有高效、环保、寿命长等优异性能，其被认为是继白炽灯和荧光灯之后的下一代照明光源。在白光 LED 中，荧光材料起着光转换的作用，是一种非常关键的材料。本论文针对 YAG:Ce<sup>3+</sup>荧光粉、YAG:Ce<sup>3+</sup>透明陶瓷以及 YAGG:Ce<sup>3+</sup>荧光材料进行了制备和发光性能方面的研究，具体内容如下：

(1) 分别以 CeF<sub>3</sub> 和 CeO<sub>2</sub> 为铈源，采用固相反应制备了一系列 YAG:Ce<sup>3+</sup>荧光粉。以 CeF<sub>3</sub> 为铈源在 1350℃ 就能制备出纯相 YAG:CeF<sub>3</sub> 荧光粉，而以 CeO<sub>2</sub> 为铈源则需要 1450℃ 才能制备出纯相 YAG:Ce<sub>2</sub>O<sub>3</sub> 荧光粉；1540℃ 制备出的 YAG:CeF<sub>3</sub> 荧光粉量子产率高达 91%，而 YAG:Ce<sub>2</sub>O<sub>3</sub> 荧光粉的只有 80%；在 150℃ 时，YAG:CeF<sub>3</sub> 荧光粉和 YAG:Ce<sub>2</sub>O<sub>3</sub> 荧光粉的发光强度分别能保持其在 25℃ 时的 85% 和 86%。

(2) 采用真空烧结法制备了 YAG:CeF<sub>3</sub> 透明陶瓷和 YAG:Ce<sub>2</sub>O<sub>3</sub> 透明陶瓷，研究了不同的粉体制备温度和陶瓷烧结温度对陶瓷样品晶相、微观结构以及透光率的影响。经过对比发现，无论是以 CeO<sub>2</sub> 为铈源还是以 CeF<sub>3</sub> 为铈源，采用 1400℃ 保温 4h 制备出的粉体在 1650℃ 真空烧结 4h 获得的 YAG:Ce<sup>3+</sup>陶瓷的透光率最高；在最佳制备条件下，1mm 厚的 YAG:CeF<sub>3</sub> 透明陶瓷在 550nm 处的透光率为 50.5%，而 1mm 厚的 YAG:Ce<sub>2</sub>O<sub>3</sub> 透明陶瓷在 550nm 处的透光率只有 45.4%。

(3) 系统地研究了 Ga<sub>2</sub>O<sub>3</sub> 含量对 YAGG:Ce<sup>3+</sup>荧光粉的荧光光谱，荧光量子产率和荧光热稳定性的影响。实验结果表明，随着 Ga<sub>2</sub>O<sub>3</sub> 含量的增加，YAGG:Ce<sup>3+</sup>荧光粉的发射光谱不断蓝移，荧光量子产率和荧光热稳定性持续下降，其中 Y<sub>3</sub>Al<sub>3.5</sub>Ga<sub>1.5</sub>O<sub>12</sub>:0.06Ce<sup>3+</sup> 的性能表现良好。Ce<sup>3+</sup> 在 Y<sub>3</sub>Al<sub>3.5</sub>Ga<sub>1.5</sub>O<sub>12</sub> 基质中的临界浓度约为 2%，临界距离约为 19Å；Y<sub>2.94</sub>Al<sub>3.5</sub>Ga<sub>1.5</sub>O<sub>12</sub>:0.06Ce<sup>3+</sup> 荧光粉的荧光热稳定性和荧光量子产率能与市售 Lu<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>:Ce<sup>3+</sup> 的媲美，远胜于市售 (Sr,Ba)<sub>2</sub>SiO<sub>4</sub>:Eu<sup>2+</sup> 的荧光热稳定性和荧光量子产率；在 1600℃ 的真空环境中 (1.5×10<sup>-3</sup>Pa) 烧结 4h 可以制备出在 530nm 处透光率大于 45% 的 Y<sub>2.94</sub>Al<sub>3.5</sub>Ga<sub>1.5</sub>O<sub>12</sub>:0.06Ce<sup>3+</sup> 透明陶瓷 (双面抛光，厚度为 1mm)，该透明陶瓷有望应用于远程激发型高显色指数白光 LED。

**关键词：**白光 LED；YAG:CeF<sub>3</sub>；YAGG:Ce<sup>3+</sup>

## Abstract

Due to its high efficiency, environmental friendliness and long lifetime, white light emitting diodes (LEDs) has been considered as a next generation lighting source after incandescent and fluorescent lamps. Playing a role in light conversion, phosphor is one of the key materials in white LEDs. In this thesis, the preparation and luminescence properties of YAG:Ce<sup>3+</sup> phosphor, YAG:Ce<sup>3+</sup> transparent ceramics and YAGG:Ce<sup>3+</sup> phosphor were studied. The main contents are as follows:

(1) A series of CeF<sub>3</sub>-doped Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (YAG:CeF<sub>3</sub>) phosphor and CeO<sub>2</sub>-doped Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (YAG:Ce<sub>2</sub>O<sub>3</sub>) phosphor were successfully synthesized by a solid-state reaction method. The pure phase YAG:CeF<sub>3</sub> phosphor can be obtained at 1350 °C, which is much lower than that of synthesizing the pure phase YAG:Ce<sub>2</sub>O<sub>3</sub> phosphor (1450 °C). The luminescence quantum yield (QY) of YAG:Ce<sub>2</sub>O<sub>3</sub> phosphor prepared at 1540 °C for 4h is just 80%, but the QY of YAG:CeF<sub>3</sub> phosphor prepared at 1540 °C for 4h is as high as 91%. At 150 °C, the luminescence intensity of YAG:CeF<sub>3</sub> phosphor and YAG:Ce<sub>2</sub>O<sub>3</sub> phosphor maintains 85% and 86% of that measured at 25 °C, respectively.

(2) Transparent YAG:CeF<sub>3</sub> and YAG:Ce<sub>2</sub>O<sub>3</sub> ceramics were successfully prepared by using vacuum sintering method. The effect of synthesis temperature of the powder and sintering temperature of the ceramics on the crystal phase, microstructure and transmittance of the ceramic samples was investigated in detail. It was found out that the transmittance of the ceramics sintered at 1650 °C for 4h by using the powders (whether YAG:CeF<sub>3</sub> or YAG:Ce<sub>2</sub>O<sub>3</sub>) prepared at 1400 °C for 4h is the highest. Under the optimum preparation conditions, the transmittance at 550 nm of the transparent YAG:CeF<sub>3</sub> ceramic (thickness of 1mm) is 50.5%, but the transmittance at 550 nm of the transparent YAG:Ce<sub>2</sub>O<sub>3</sub> ceramic (thickness of 1mm) is only 45.4%.

(3) The effect of Ga<sub>2</sub>O<sub>3</sub> concentration on excitation and emission spectra, QY and luminescence thermal stability of YAGG:Ce<sup>3+</sup> phosphors were investigated in detail. With the increase of Ga<sub>2</sub>O<sub>3</sub> concentration, the emission spectra of YAGG:Ce<sup>3+</sup> show a continuous blue

shift, its QY and luminescence thermal stability continuously decline.  $\text{Y}_{2.94}\text{Al}_{3.5}\text{Ga}_{1.5}\text{O}_{12}:0.06\text{Ce}^{3+}$  shows relatively good performance. The critical concentration and critical distance of  $\text{Ce}^{3+}$  ions in  $\text{Y}_3\text{Al}_{3.5}\text{Ga}_{1.5}\text{O}_{12}$  host is about 2% and 19 Å, respectively. The QY and luminescence thermal stability of  $\text{Y}_{2.94}\text{Al}_{3.5}\text{Ga}_{1.5}\text{O}_{12}:0.06\text{Ce}^{3+}$  phosphor are just a little worse than those of a commercial  $\text{Lu}_3\text{Al}_5\text{O}_{12}:\text{Ce}^{3+}$  phosphor but much better than those of a commercial  $(\text{Sr},\text{Ba})_2\text{SiO}_4:\text{Eu}^{2+}$  phosphor. The transparent  $\text{Y}_{2.94}\text{Al}_{3.5}\text{Ga}_{1.5}\text{O}_{12}:0.06\text{Ce}^{3+}$  ceramic can be prepared at 1600 °C for 4 h under vacuum ( $1.5 \times 10^{-3}$  Pa) environment, its transmittance at 530 nm is more than 45% (double faces polished, thickness of 1 mm). The transparent ceramic is expected to be applied to the remote excitation type high color rendering index white LEDs.

**Keywords:** White LEDs;  $\text{YAG}:\text{CeF}_3$ ;  $\text{YAGG}:\text{Ce}^{3+}$

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## 第一章 绪论

纵观人类照明光源的发展历史，在很长一段时间里，篝火、油灯、蜡烛是人类的主要照明光源。直到 19 世纪 80 年代初，商品化的白炽灯使人类从火光照明时代跨入了电气照明时代。继白炽灯之后，荧光灯逐渐变成了照明领域的主流光源。随着科技的不断进步，1996 年日本日亚化学工业株式会社（日亚化学）成功地开发出了基于蓝色发光二极管（light emitting diode, LED）的白光 LED，从此白光 LED 很快商品化并得到了快速发展。如图 1.1 所示，目前白光 LED 已经被广泛地应用于室内照明（图 1.1a）、户外照明（图 1.1b）、显示器背光照明（图 1.1c）和交通工具照明（图 1.1d）等领域。



图 1.1 白光 LED 的主要应用领域：室内照明（a）、户外照明（b）、背光照明（c）和汽车照明（d）

Fig. 1.1 Lighting application fields of white LEDs: indoor lighting (a), outdoor lighting (b), backlighting (c) and automotive lighting (d)

### 1.1 白光 LED

#### 1.1.1 白光 LED 简介

通常所谓的白光 LED 并不是指直接发白光的单个 LED，而是指以多个不同颜色的



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